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In-situ torrefaction and spherical pelletization of partially pre-torrefied hybrid poplar

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ABSTRACT

A novel compacted, spherical, torrefied biomass pellet is discussed. Pellets were pressed in a heated mould and die, and torrefied in-situ (at 280 °C) from both untorrefied and pre-torrefied (at 250 °C) hybrid poplar sawdust prior to pelleting. Spherical pellets were successfully produced from both input materials. The calorific value of these pellets, referred to as "Q'Pellets", is 20.78 MJ kg^{-1}–21.60 MJ kg^{-1} on a moisture and ash free basis, which compares favourably with lignite coal. The ash content of Q'Pellets was as high as a mass fraction of 2.07%–5.08%. Q'Pellets are demonstrated to be uniquely durable, as they did not abrade in a tumbling can test or fracture in an impact resistance (drop) test.

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1. Introduction

Most biomass pellets, known as "white" pellets, are made from raw biomass using an extrusion process. Woody biomass, such as poplar, willow and grasses, such as hemp, switchgrass, miscanthus are examples of feedstocks. To produce white pellets, raw biomass is typically hammer-milled to pass a 4 mm screen, and conditioned to a moisture content of mass fraction 8%–12% [[1], p. 57] prior to pelletization. Pelletization involves a press that forces material through a friction-heated extrusion die, which has multiple holes of approximately 6 mm diameter. Cylindrical pellets approximately 2–5 cm long with smooth sides and rough ends are thereby formed. The gross calorific value (GCV) and energy density of woody white pellets are approximately 19.8 MJ kg^{-1}–20.7 MJ kg^{-1} (dry basis) and 8.7 GJ m^{-3}–11.4 GJ m^{-3}, respectively [[1], p. 56–57]. Fig. 1 shows an example of typical white cylindrical biomass pellets.

White pellets have a number of deficiencies that limit their acceptance in a variety of commercial jurisdictions. For example, Duncan et al. [2,3] have identified a number of problems that prevent the adoption of biomass pellets as a fuel for large scale thermal electricity generation. These have been re-interpreted as follows:

a) Low energy density

Wood has a low calorific value compared to coal. For example, lignite and anthracite contain 20 MJ kg^{-1} and

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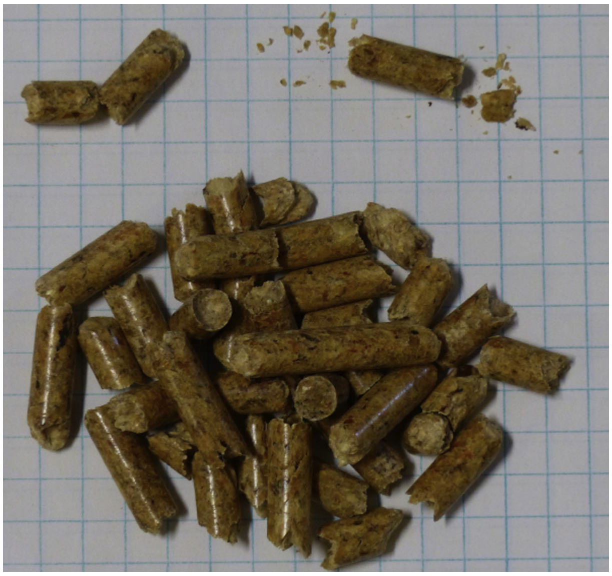

Fig. 1 – Modern cylindrical biomass pellets. Top-left: usual mode of fracture. Top-right: fines produced by scraping a fingernail across the end of the pellet. Bottom: bulk pellets. Grid paper is 6.35 mm for scale.

25 MJ kg^{-1} to 30 MJ kg^{-1}, respectively, whereas wood pellets contain only 17 MJ kg^{-1}–19 MJ kg^{-1} [[2], p. 2–3]. Thermal generating stations are designed to operate optimally with a specific fuel, and if substituted with a fuel that is "off-specification" then thermal efficiency will be altered.

b) Cost of transportation

Raw logs and wood pellets have high moisture content and low packing density compared to coal. Thus, it is comparatively expensive to transport biomass.

c) Storage

Biomass is hydrophilic; if left in an uncontrolled atmosphere will achieve equilibrium moisture content of mass fraction 4% to 25% [[1], p. 143] (depending on relative humidity) where higher moisture content correlates with lower energy density (i.e. lower heating value). White pellets that become sufficiently moist will disintegrate. Pellet piles can experience self heating due to microbial activity or moisture absorption, ultimately leading to spontaneous combustion [[1], p. 164–167]. Thus, storage of biomass in large quantities can be difficult and dangerous.

d) Grinding

Hemicellulose and cellulose, held together with lignin, form a rigid chemical structure that gives wood its strength. Where coal is brittle, biomass is malleable and "stringy". Biomass material adheres to the ball bearing-type environment found in typical coal pulverizers. Thus, woody biomass presents unwanted side effects to existing infrastructure.

e) Mechanical durability and fines production

Modern wood pellets have exposed ends, which flake and produce fines and dust. Airborne dust is a workplace health concern, and an excess of suspended particles in the air can lead to dangerous dust explosions. Fines production is a risk when transporting and handling wood pellets. Fig. 1 presents evidence of the typical issues with durability and fines.

1.1. *Benefits of torrefaction*

Torrefaction is a mild pyrolysis heat treatment that is accomplished in the temperature range 200 °C–300 °C. Torrefaction is performed in an inert atmosphere, often by nitrogen gas (N_2) flood, in order to prevent combustion. During the torrefaction process molecules are released in gaseous and condensable vapour forms, such as carbon mono- and dioxides, water vapour, acetic acid, and various other volatile organic compounds (VOCs). The result derived from torrefaction is characterized by an increase in energy density, decrease in particle size, and darkening the colour of the biomass.

There exists a prevalent notion in the biomass literature that states that the heating rate during torrefaction must not exceed 50 °C min^{-1} [4–6]. Other research has been conducted with heating rates in excess of 50 °C min^{-1} and no ill effects were noted [7]. Steam treatment of biomass as a means of torrefaction routinely features cool material inserted into a pre-heated vessel and rapid heating rates [8].

Given the five issues noted above, torrefaction addresses these through an increase in energy density and associated decrease in transportation costs, safer storage and an increase in friability.

Off-gassing molecules constitute roughly 30% the mass of the original sample, but only 10% the energy thereof. As such, the remaining solids contain 90% of the energy in only 70% of the original mass, and the ratio of energy density of the torrefied biomass compared to the original biomass is significantly increased to approximately 1.28 [[5], p. 94]. The energy density of torrefied biomass is similar to that of coal, thereby enabling co-firing or generating station conversion.

The breakdown of hemicellulose reduces the presence of hydroxyl (OH–) groups that absorb water molecules through hydrogen bonding, and non-polar molecules are formed that prevent the condensation of water. The hydrophobicity of torrefied biomass reduces its equilibrium moisture content to a mass fraction of 3% (compared with approximately 8.5% for white pellets) [[5], p. 119], and reduces self-heating through water absorption. Torrefaction also removes most of the chemically and biologically active solid, liquid and gaseous products, thereby significantly reducing the risk of spontaneous combustion [[5,9], p. 117].

Raw biomass exhibits plastic behaviour during grinding, while torrefied biomass is brittle and does not adhere to, say, pulverizer balls used to grind coal. The electrical energy required to grind biomass to a particle size suitable for co-firing with coal is reduced by up to an order of magnitude when the biomass is torrefied [[5,10], p. 116].

Torrefaction of biomass does not mitigate the production of fines or the danger of dust explosions. Material that is torrefied features reduced particle size; however, it becomes brittle and is more likely to produce dust. Perhaps more significantly, torrefied biomass has a low moisture content and high reactivity, which make it more prone to dust explosions compared to coal [[5], p. 118]. In this regard, torrefaction itself is not the panacea to the problems associated with white biomass pellets.

1.2. *The Q'Pellet*

A novel torrefied biomass pellet aimed at overcoming the challenges of white pellets by building on the benefits of torrefaction, and solving the issues of mechanical durability and fines production was originally described by Duncan et al. [2,3]. It is spherical, which is a first in the pelletized biomass industry (see Fig. 2). The spherical shape eliminates both the exposed, dusty ends and tendency for radial fracture experienced by white and torrefied cylindrical pellets. Q'Pellets are 12.7 mm diameter such that they are roughly equal in volume to cylindrical pellets.

In Ref. [2], hybrid poplar sawdust was heated in a mould and die, torrefied in-situ, and compressed to produce spherical pellets. The present work focuses upon improvement to that process through methodology improvement and feedstock material pre-treatment. It is demonstrated herein that woody biomass can be torrefied in a mould and die that is preheated to torrefaction temperatures, similar to the rapid heating rates used in steam treatment. The time required for dry torrefaction is greatly reduced compared to beginning the process at room temperature. It is also demonstrated that woody biomass that has been previously torrefied can be reheated in-situ and pressed to form pellets.

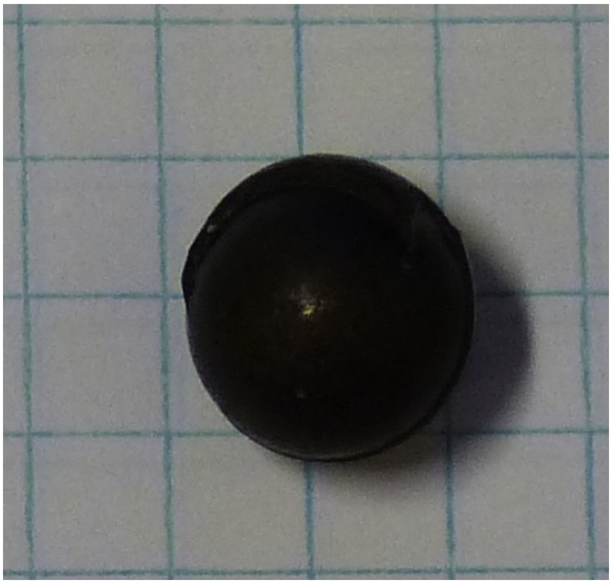

Fig. 2 – The Q'Pellet. Grid paper is 6.35 mm for scale.

2. Materials and methods

2.1. *Material selection and preparation*

Pellets were created using hybrid poplar (*populus hybrids*). Poplar was selected for its ideal properties as an energy crop—it can be harvested in short rotations on marginal land and requires very little maintenance. Eastern Ontario Model Forest (EOMF), located near Kemptville, Ontario, supplied the hybrid poplar sample. The sample was harvested, de-barked, and chipped by EOMF personnel in 2009, dried to ambient moisture content at room temperature in a perforated bag at Queen's University in Kingston, Ontario, and hammer milled (Pellet Pros model PP H 1000E) to pass a 3.55 mm screen. The sample was carefully stored away from other biomass at ambient temperature and moisture content to be used as needed.

Hammer milled material was sieved by hand. Material that passed US No. 16 and No. 30 screens was used in a mass ratio of 40:60, respectively, while material passing a US No. 50 screen was rejected, in order to ensure ideal particle binding, which has been demonstrated to create a “strong” pellet [2,3].

Material was dried at 103 °C ± 1 °C in approximate accordance with ASTM Method E871.23220 [11]. An analytic balance (Cole–Parmer RK-10000-16) was used to determine the initial mass of the sample, and repeated in 1 h intervals. The sample was determined to be sufficiently dry when the mass loss between weighings was less than 0.2%. Two samples were dried: one for use in the batch torrefier and one for producing pellets from raw material, with each of mass approximately 30 g. Samples were stored in air-tight containers to prevent reabsorption of moisture.

The desired mass of each torrefied pellet was 1.5 g–1.6 g, which was achieved by using different amounts of raw or pre-torrefied material based on mass lost during pre-torrefaction and expected mass loss during in-situ torrefaction. Pellets made from raw material required 2.00 g; pellets made from pre-torrefied material required 1.75 g. Individual samples were stored in air-tight glass vials.

2.2. *Batch torrefier*

A torrefaction vessel was used to test the effects of torrefying material ex-situ prior to pelleting. A mild torrefaction treatment was used so that torrefaction could be completed in-situ during pelleting. The design was derived from Bergman et al. [4] and constructed by Duncan [2]. The vessel used a heating tape wrapped around an aluminium chamber (diameter 10.80 cm, length 30.48 cm) enclosed in Fiberglass™ insulation and aluminium shim. A metal wire mesh basket held the dry sample (29 g) to be torrefied. No effort was made to capture or analyse the off-gasses.

The temperature within the torrefaction chamber was measured using pre-calibrated J-type thermocouples (OMEGA Engineering). The heating tape was controlled using a benchtop digital controller (OMEGA CSi8DH) under PID control. The time–temperature history was recorded in 5 s increments using a data acquisition module (OMEGA OM-DAQ-USB-2401).

The raw, dry sample was sealed within the vessel and the system flushed with N_2 at a flow rate of 40 mL min^{-1} for 10 min. An N_2 flow rate of 20 mL min^{-1} maintained an inert atmosphere inside the torrefier and carried away off-gasses, while a torrefaction time and temperature (according to the terminology of Ref. [4]) of 30 min and 250 °C, respectively, ensured only partial torrefaction of the sample. The sample was cooled in the chamber under constant nitrogen flow before being removed and placed in an air-tight glass jar.

2.3. *Pelleting apparatus and procedure*

Compressing biomass between two hemispherical mould faces was found to result in a pellet with a weak equatorial plane, which is prone to fracture. The mould used to create the Q'Pellet was specially designed, Duncan et al. [12] (International Patent Publication Number WO 2012/106801 A1) to increase pellet strength. Two mould halves, made of M2 tool steel and housed in a cylindrical sleeve, formed the spherical mould cavity and compression channel for the die (see Fig. 3). The axis along which the die was compressed was parallel to, but not coincident with, the central axis of the mould. This caused the material to reorient and interlock during compression, thereby eliminating the weak equatorial plane. A sketch of the assembled mould cross section is shown in Fig. 4. A Carver Model C press with manual hydraulic pump was used to compress the material into spherical pellets.

The PID controller maintained the desired torrefaction temperature using a heating tape that was wrapped around the Q'Pellet mould. A K-type thermocouple was inlaid into the mould adjacent to the internal cavity and secured using high temperature cement. Time–temperature history for each pellet was recorded in 5 s increments.

The mould was pre-heated to 280 °C. Material sufficient to make one pellet was funnelled into the mould and the die placed atop it. Material was heated and torrefied for 3 min before the pellets were compacted. Compaction of each pellet took 15 s–20 s, and pellets were held in the mould under maximum compression for 1 s. Maximum compression was defined by the die coming flush within the mould such that the cavity was perfectly spherical, and typically occurred around 1.5–2.0 tonnes.

Pellets were extracted by removing the mould from the sleeve and splitting the mould halves apart. Pellets, including any flashing or loose material, were stored in air-tight vials to cool. Once cooled to room temperature, the pellets were massed both with and without flashing in order to determine mass loss due to torrefaction and mass that was mechanically excluded from the pellet. Flashing was removed by vigorously shaking individual pellets in a glass vial for 15 s.

Ten Q'Pellets were made in this experiment. Five were made from raw material, and five were made from pre-torrefied material.

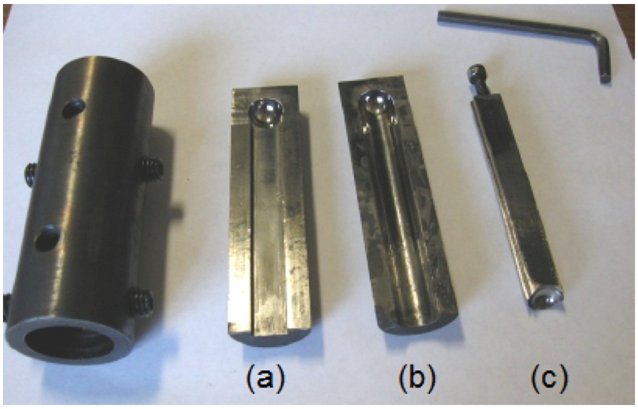

Fig. 3 – The mould halves (a and b) and the die (c).

2.4. *Mould release properties of torrefaction residues*

When the mould and die were initially machined, the inner surfaces had a slightly rough finish which was, through experimentation, deemed unsuitable for producing pellets with a smooth outer surface that was typical of those invented by Duncan [2]. A felt-tipped Dremel tool was used with diamond paste together with high temperature polish paste to smooth the inner surfaces to a mirror finish. The mould and die were subsequently polished between each pelleting run. Pellets were successfully formed, but occasionally stuck in the mould and were difficult to remove.

Consideration was given to using a lubricant or mould-release agent, but the ability to make pellets exclusively from raw material, with no binders or other additives, was

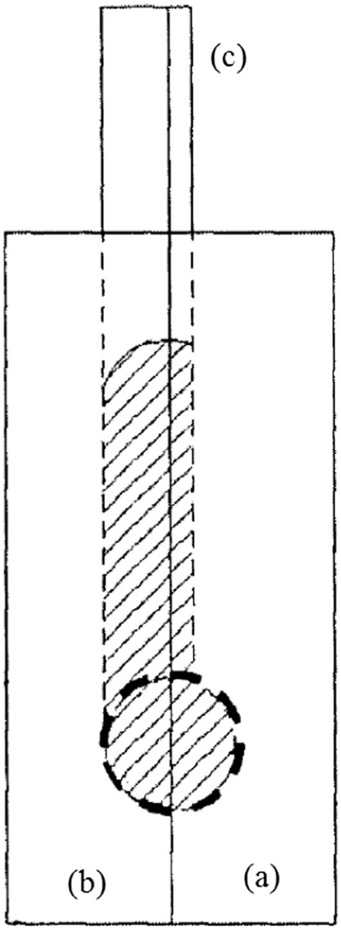

Fig. 4 – Cross-section of the assembled mould and die. Hatching shows space occupied by material during in-situ torrefaction. The mould halves (a and b) and the die (c) are labelled as in Fig. 3.

Table 1 – Experimental methods and apparatus used by SGS North America in ultimate and short proximate analysis.

Test	Method	Equipment
Total moisture	ASTM D4442 (Method A)	Despatch oven
Ash	ASTM D1102	Thermolyne muffle furnace
Calorific value	ASTM D5865	IKA C5000 calorimeter
Sulphur	ASTM D4239 (Method A)	LECO SC632 sulphur analyzer
Carbon	ASTM D5373	LECO TruSpec CHN analyzer
Hydrogen	ASTM D5373	LECO TruSpec CHN analyzer
Nitrogen	ASTM D5373	LECO TruSpec CHN analyzer
Oxygen (by difference)	ASTM D5373	–

deemed to be a required outcome. By virtue of continuous use, the condensable liquid vapours and solid components released through off-gassing during the in-situ torrefaction process coated the inner surfaces of the mould and die (a colour change was observed), and the polishing step was omitted between subsequent pelleting runs. The condensed components accumulated to form an anti-stick coating inside the mould and die. This facilitated mould release, and bears the obvious side benefit that the time consuming, labour intensive polishing step can be omitted.

Increased ease of mould release with continued mould use is also observed in the injection moulded polymer industry. Mould release agents are used to prevent parts becoming stuck and to facilitate part release in new moulds [13]. However, mould release agents are no substitute for proper mould design [14]. A mould which has been properly designed and manufactured typically develops an intrinsic property that causes parts to release cleanly without mould release agents after a reasonable “break-in” period [15]. This phenomenon may lend insight into the mould release properties of torrefaction residues.

2.5. *Mechanical durability testing*

Abrasion resistance is a common measure of mechanical durability. Coal (ASTM D441-07(2012)) is tested by tumbling 1000 g of sieved material in a baffled jar at $0.\overline{6}$Hz for 1 h and determining the final particle size distribution. Biomass pellets (CEN/TS 15210-1 [16]) are tested by rotating a single central baffle at $0.8\overline{3}$Hz for 10 min through 500 g of pellets in a dust-tight box and determining the mass of fines that pass a 3.15 mm screen. These methods were inappropriate for testing the abrasion resistance of a single pellet, so that a hybrid method was developed which was intended to represent the most harsh aspects of either standard noted previously. A plastic tumbling can (diameter 8 cm, length 7 cm) with three moulded baffles was used to tumble a single pellet at 1 Hz for 1 h. Twenty glass marbles (average mass 5.3 g) were included in the tumbling can to represent the effects of other pellets on the test pellet. Mechanical durability was calculated as recommended by the CEN standard, according to.

$$MD = \frac{m_{\mathrm{final}}}{m_{\mathrm{initial}}} \times 100\% \tag{1}$$

where MD is the mechanical durability, m_{final} is the mass of the pellet after tumbling, and m_{initial} is the mass of the pellet before tumbling. Results are presented in Section 3.1.

Impact resistance was also used to assess the mechanical durability of pellets. Richards [17] introduced the Impact Resistance Index (IRI) as a means to compare the durability of charcoal fuel briquettes. Briquettes were repeatedly dropped (N) from a height of 2.0 m onto a concrete floor, and the number of pieces (n) the briquette broke into was recorded. IRI was calculated as

$$IRI = \frac{100 \times N}{n} \tag{2}$$

Li and Liu [18] used IRI to assess biomass briquettes, and used $N = 2$ as a standard to facilitate comparison. In this case, the maximum possible IRI is 200. Individual Q'Pellets were dropped twice from a height of 2.0 m ± 0.1 m in order to determine IRI. Results are presented in Section 3.1.

2.6. *Ultimate and proximate analysis*

Ultimate analysis is the assessment of a hydrocarbon fuel based on its elemental composition [[5], p. 73–75]. Relative composition of carbon (C), hydrogen (H), oxygen (O), nitrogen (N), and sulphur (S) are expressed as mass fractions in %. Ash (inorganic matter), moisture content, and Gross Calorific Value (GCV) are determined by proximate analysis. Torrefaction increases the carbon composition and decreases the hydrogen and oxygen composition of a fuel, thereby increasing its energy density. Nitrogen and sulphur are undesired elements.

Q'Pellets were sent to SGS North America's Minerals Division lab in South Holland, IL, a fee-for-service analytical lab. A “short” proximate analysis was performed due to the small mass of individual pellets. A sample of untorrefied hybrid poplar was also analysed for comparison. The analysis was conducted according to the specifications presented in Table 1.

Results are presented in Section 3.2.

3. Presentation and discussion of results

3.1. *Pellet durability*

All pellets achieved mechanical durability of 100% in the tumbling can test (according to Equation (1)). The final mass of each pellet was equal to, or, surprisingly slightly larger than, the initial mass of that pellet.

The creation of a pellet that does not abrade was an industry first for biomass pellets. While the abrasion test does not strictly follow an established ASTM or CEN standard, the tumbling can test performed was sufficiently harsh to represent the forces a pellet would be expected to experience during shipping and handling. Although the can used for tumbling was a soft plastic (rather than metal or porcelain), the inclusion of glass marbles replaced the hard outer surface with a hard inner surface. In some cases the final mass actually exceeded the initial mass by a very small amount. The plastic of the rotating drum and baffles is rough to the

Table 2 – Ultimate and proximate analysis on an "as received" basis. GCV≡ Gross Calorific Value (Higher Heating Value); C≡ Carbon; H≡ Hydrogen; N≡ Nitrogen; S≡ Sulphur; O≡ Oxygen; AVG≡ Average; STD≡ Standard Deviation (of a sample).

Source	Pellet #	Density (kg m^{-3})	Moisture (%)	Ash (%)	GCV (MJ kg^{-1})	C (%)	H (%)	N (%)	S (%)	O (%)
Untreated	1	1293	2.51	2.07	20.18	53.43	5.71	<0.01	0.01	36.27
	2	1262	3.09	2.29	20.19	53.86	5.59	<0.01	0.02	35.15
	3	1292	2.71	2.36	20.23	53.93	5.69	<0.01	0.02	35.29
	4	1289	2.82	2.70	20.16	54.26	5.64	<0.01	0.02	34.56
	5	1321	2.87	2.38	20.19	53.56	5.69	<0.01	0.01	35.49
	AVG	**1292**	**2.80**	**2.36**	**20.19**	**53.81**	**5.66**	–	**0.02**	**35.35**
	STD	**21**	**0.21**	**0.23**	**0.02**	**0.33**	**0.05**	–	**0.01**	**0.62**
Pre-torrefied	6	1275	2.61	2.92	20.12	54.01	5.31	0.42	0.01	34.72
	7	1254	3.10	5.08	19.83	52.87	5.02	0.44	0.02	33.47
	8	1271	2.79	3.00	19.58	53.22	5.30	0.38	0.03	35.28
	9	1271	2.60	4.35	16.94	49.06	4.49	0.39	0.02	39.09
	10	1279	2.22	2.92	20.21	53.16	5.30	0.38	0.02	36.00
	AVG	**1270**	**2.68**	**3.48**	**19.94**	**53.32**	**5.23**	**0.41**	**0.02**	**34.87**
	STD	**11**	**0.37**	**1.07**	**0.29**	**0.49**	**0.14**	**0.03**	**0.01**	**1.07**
Raw poplar		–	7.32	2.21	16.83	46.57	5.24	0.45	0.02	38.19

touch, and subject to abrasion itself during the tests (especially when glass marbles are included). The additional mass picked up by the pellets was likely the small amount of mass lost by the tumbling can.

Following the tumbling can test, impact resistance tests were conducted and the IRI calculated according to equation (2). Each pellet remained fully intact throughout the drop tests, and a value of $n = 1$ for each pellet yielded an IRI $= 200$. It was found that Q'Pellets bounce when they hit the ground, which is unlike white pellets. Q'Pellets were allowed to bounce 3–7 times each. Further, the pellets were thrown towards the ground and easily bounced back up into the authors' hands without any discernable change to their physical appearance.

Achieving high abrasion and impact resistance is crucial to solving transportation and fines issues associated with modern biomass pellets. The tumbling can test represents the constant bumping and rubbing pellets would experience during transportation by truck or rail. The impact drop test simulates materials handling stages, such as loading pellets into a truck bed or a storage silo.

3.2. *Ultimate and proximate analysis*

Results of the ultimate and short proximate analysis are presented in Table 2 on an "as received" basis and in Table 3 on a moisture free basis. Average values are presented for pellets made from untreated material and pre-torrefied material.

Pellet #9 is identified as an outlier. While the pelleting process was the same for all pellets made for these tests, the composition of the pellet is atypical. It is not clear why this is the case. We offer the following as possible reasons: a) the temperature of the mould was measured using a thermocouple that was inserted into the mould wall. The thermocouple leads were routinely bent and unbent to fit the mould under the press, and thus the thermocouple wire became frayed, which caused the thermocouple to short-circuit and record an incorrect temperature, as was observed occasionally in previous experiments but was not noticed in this particular case; b) room temperature material was loaded into the pre-heated die, while the mould cavity was filled with air. It is possible that, due to the presence of a localized interstitial

Table 3 – Ultimate and proximate analysis on a moisture free basis. GCV≡ Gross Calorific Value (Higher Heating Value); C≡ Carbon; H≡ Hydrogen; N≡ Nitrogen; S≡ Sulphur; O≡ Oxygen; AVG≡ Average; STD≡ Standard Deviation (of a sample). Note: GCV is given on moisture and ash free basis for more universal comparison.

Source	Pellet #	Ash (%)	GCV (MJ kg^{-1})	C (%)	H (%)	N (%)	S (%)	O (%)
Untreated	1	2.12	21.15	54.81	5.86	<0.01	0.01	37.20
	2	2.36	21.34	55.58	5.77	<0.01	0.02	36.27
	3	2.42	21.31	55.43	5.85	<0.01	0.02	36.28
	4	2.77	21.34	55.83	5.81	<0.01	0.02	35.57
	5	2.45	21.31	55.14	5.86	<0.01	0.01	36.54
	AVG	**2.42**	**21.29**	**55.36**	**5.83**	–	**0.02**	**36.37**
	STD	**0.23**	**0.08**	**0.40**	**0.04**	–	**0.01**	**0.59**
Pre-torrefied	6	3.00	21.30	55.46	5.45	0.44	0.01	35.64
	7	5.25	21.60	54.56	5.18	0.46	0.02	34.53
	8	3.08	20.78	54.75	5.45	0.40	0.03	36.29
	9	4.47	18.21	50.37	4.61	0.41	0.02	40.12
	10	2.99	21.31	54.36	5.42	0.39	0.02	36.82
	AVG	**3.58**	**21.25**	**54.78**	**5.38**	**0.42**	**0.02**	**35.82**
	STD	**1.11**	**0.34**	**0.48**	**0.13**	**0.03**	**0.01**	**0.99**
Raw poplar		2.38	18.61	50.25	5.65	0.49	0.02	41.21

oxygen pocket, a small combustion event occurred at the beginning of torrefaction. Data for Pellet #9, we believe, is not representative of the Q'Pellet production process, and is therefore excluded from the calculation of average values for pellets made from pre-torrefied material in Tables 2 and 3.

Pellets made from untreated and pre-torrefied biomass have an average energy density of 20.19 MJ kg^{-1} and 19.94 MJ kg^{-1}, respectively. This is significantly higher than that of raw hybrid poplar, and comparable to that of lignite coal. This correlates with the increase in carbon and decrease in oxygen content through torrefaction. The change in elemental hydrogen is small.

3.3. *Comparison of untreated and pre-torrefied pellets*

Pellets made from untreated sawdust exhibit less variability than those made from pre-torrefied material. Tables 2 and 3 indicate that pellets 1–5 exhibit a smaller standard deviation for all averaged values than do pellets 6–8 and 10.

The ash content of pre-torrefied material is larger on a weight percentage basis. This is due to the increased volatile mass loss through pre-torrefaction, and the requirement to use more total material to form each pellet. The calorific value of pre-torrefied pellets is therefore lower than untreated pellets because of increased ash content, as shown by roughly equal calorific values on a moisture and ash free (MAF) basis.

The difference in nitrogen content between untreated and pre-torrefied pellets is significant. All untreated pellets have nitrogen content below the detection limits of the tests performed, whereas pellets created from pre-torrefied material have an average nitrogen mass fraction 0.41%, similar to raw hybrid poplar. Neither of these cases is without precedent. Prior research [6,19] has shown that torrefaction in nitrogen atmosphere does not cause a decrease in nitrogen content. Torrefaction through steam treatment, performed without a nitrogen atmosphere, has been observed [8] to cause a slight decrease in nitrogen content. It is hypothesized that the presence of a nitrogen atmosphere during torrefaction prevents the removal of nitrogen molecules from the sample.

3.4. *Spring-back and over-pressing*

The height, width, and depth of each pellet were measured using micrometres. The width and depth were typically equal within measurement error. The aspect ratio (AR) of each pellet was calculated as the ratio of its height (measured in the direction of compression) to width (equatorial plane), and was typically in the 1.04–1.09 range. This represents a “spring-back” effect, where the pellet pushed the die back up out of the mould once the hydraulic press was released.

Pellets that exhibited spring-back were not spherical. As such, the volume of the pellet was calculated as the volume of an ellipsoid rather than a sphere. Pellets that exhibit spring-back have larger volumes, and thus lower mass densities, than pellets that remain perfectly spherical. In other pelleting trials using the apparatus described above, a method of “over-pressing” was used to lessen the effects of spring-back. In over-pressing trials the die was compressed farther into the mould (i.e. beyond flush), such that the aspect ratio at the time of pressing was less than one. When the pressure was released, the pellet was able to spring-back to a very nearly spherical shape, with an aspect ratio of unity and lower volume than if it was ellipsoid.

The spring-back effect may be an advantage. During transportation of pellets it is the bulk density which is of key importance. Bulk density is based on mass density, and on the packing factor of the object (the volume fraction of space occupied by objects). Depending on their relative dimensions, ellipsoids can have a significantly higher random packing factor than perfect spheres. In this case, the spring-back of pellets may decrease the mass density, but ultimately increase the bulk density. Further experimentation would reveal the optimal balance between over-pressing and spring-back.

3.5. *Impact and future work*

A number of interesting results manifest from the methodology used to torrefy material in-situ for the production of Q'Pellets. Material that has been inserted into a pre-heated die is rapidly heated to torrefaction temperatures, in contradiction to much of the literature which states torrefaction rates must not exceed 50 °C min^{-1} (see Section 1.1). This torrefaction also occurs without the use of an inert atmosphere due to the confined environment; that is, the mould and die act to mechanically separate air from the sample and off-gassing produces a sufficient back-pressure to prevent any air or oxygen from flowing into the apparatus.

A number of technical specifications and certification systems have been developed to standardize biofuel quality. The ENplus certification system, based on fuel specifications in CEN/TS 15234:2006 [20] and CEN/TS 14961:2005 [21], is the principal certification system in Europe and is considered herein due to its strict fuel quality requirements. The ENplus system has three fuel quality levels [1]: ENplus A1 is the most strict and represents pellets for use in small boilers by private end users; ENplus A2 is slightly relaxed in its ash, GCV, and nitrogen content requirements and is appropriate for use in mid-scale boilers by commercial users; EN B certification further relaxes specifications and is considered appropriate for use in industrial applications. This fuel specification is intended to apply to white pellets, but as a suitable benchmark for torrefied pellets is not available, the ENplus system is used to evaluate the Q'Pellet.

The Q'Pellet does not meet the ENplus A1 specifications for ash content (mass fraction 0.7% dry basis). The pellets made from pre-torrefied material also exceed the nitrogen content maximums (mass fraction 0.03% dry basis). These excesses are due to the feedstock selected rather than the torrefaction and pelleting methodology. Where a typical deciduous wood (according to CEN/TS 14961:2005) has an ash content of mass fraction 0.3% (dry basis), poplar has a typical ash content of mass fraction 2.0%. Similarly, a typical deciduous wood has a nitrogen content of mass fraction 0.1% (MAF basis), and poplar has a typical nitrogen content of mass fraction 0.4% [21]. The poplar feedstock used to make Q'Pellets had ash and nitrogen contents of mass fraction 2.38% (dry basis) and 0.49% (dry basis), respectively. It is reasonable to extrapolate that if a feedstock was used that had lower ash and nitrogen contents than poplar, then the ash and nitrogen contents of the produced Q'Pellets should be significantly lower and more in line with top quality fuel specifications. Future work should involve the testing of the developed pelleting method using different feedstock materials.

The Q'Pellets produced from untreated hybrid poplar meet the EN B fuel specification, thereby making them appropriate for industrial use. Three principal industries use coal: thermal power generation, cement, and steel.

Many thermal generating stations burn coal in order to produce electricity. Biomass can be co-fired with, or burned instead of coal, such as at Ontario Power Generation's Atikokan Generating Station (GS) in Ontario, Canada. Thermal power stations typically have strict limitations on ash content and quality in order to prevent fouling of boilers. The biomass fuel specification for Atikokan GS dictates that ash content should be below a mass fraction of 1.5% (as received) [22]. The Q'Pellet, which was made from poplar, may not be appropriate for use in thermal power generation.

The cement industry burns coal and petroleum-coke to produce the heat required for making clinker, and is much more relaxed in their ash content restrictions. Ash in a cement kiln typically falls into the clinker and is absorbed without reduction in product quality or fouling of the kiln. At Lafarge's cement plant in Bath, Ontario, biomass fuels with an ash content as high as a mass fraction of 5%can be fired without adverse effects. Nitrous oxide (NO_x) emissions, however, are a sensitive issue with regulators. The nitrogen content in biomass fuel must be equal to or less than the nitrogen content of the coal or petcoke it replaces, so that no increase in NO_x emissions is expected. This limits nitrogen content to a mass fraction of approximately 1.4%. As such, Q'Pellets produced from either untreated or pre-torrefied material can be used as a substitute for coal in the cement industry.

The steel industry also uses coal in blast furnaces as a reducing agent for iron ore and as a combustion fuel to provide heat to the reduction reaction. Torrefied biomass is suitable for co-firing with pulverized coal in a blast furnace [23], and some research indicates that it may also be suitable as a reducing agent [24]. Based on the desire of the steel industry to reduce its carbon intensity, the Q'Pellet may be used in either of these roles.

4. Conclusions

The Q'Pellet achieves best-in-industry durability for biomass pellets. All pellets exhibited complete abrasion and impact resistance. The tumbling test represents the constant abrasive forces a pellet will experience during transportation, and the impact test represents handling stages where pellets are dropped during loading. The durability of this pellet will greatly reduce the production of dust and fines during transportation and material handling.

The energy density of the Q'Pellet is 19.58 MJ kg^{-1} to 20.23 MJ kg^{-1} on an as received basis, and 20.78 MJ kg^{-1}–21.60 MJ kg^{-1} on a MAF basis. This is comparable to lignite coal, and makes the Q'Pellet an ideal candidate for co-firing with lignite or as fuel for a converted lignite-fired generating station. The ash content is mass fraction 2.07%–5.08%, which is higher than most fuel specifications. However, the ash content for raw hybrid poplar is mass fraction 2.21%, which indicates that if a lower ash feedstock were used the Q'Pellet could be within fuel specifications.

It is possible to make pellets from either untreated poplar sawdust or pre-torrefied material. This increases the flexibility of the production process by allowing the torrefaction and pelletization stages to be decoupled.

Further work in this area will involve scale-up to a continuous pelleting process that also embraces different feedstock materials.

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